

PHILIP MORRIS, INC.
RESEARCH AND DEVELOPMENT DEPARTMENT

PROJECT 21-1100

QUANTITATIVE DETERMINATION OF PROPYLENE GLYCOL ON CIGARETTE
FILLER

Completion Report

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I. Introduction and Summary

A method for the quantitative determination of propylene glycol on cigarette filler was developed. The method employs a gas chromatographic analysis of the methanol extract of a single cigarette.

The method could not be readily extended to the quantitative analysis of glycerol because of the great difference in the vapor pressures of propylene glycol and glycerol at the temperatures required for the analysis of propylene glycol.

II. Experimental Procedure, Results and Discussion

A series of experiments was performed to establish the retention times of propylene glycol and glycerol when chromatographed on columns of various phases at different temperatures. The column best suited for the separation of these two compounds was a column of Ucon/Chromosorb (2:8 w/w).

At temperatures best suited for the chromatography of glycerol, the propylene glycol emerged with the methanol solvent and at lower temperatures the retention time of glycerol was too great.

The above difficulty was overcome by flow-programming the two compounds at 168 and 180°C. At these temperatures, using selected flow rates, it was possible to elute propylene glycol and glycerol at satisfactory times.

These temperatures caused a peculiar shape in the glycerol peak which made the determination of area impractical. Higher temperatures were not satisfactory for the measurement of the propylene glycol peak.

The method was applied to the determination of propylene glycol alone because of these difficulties. The procedure finally adopted is reported in the Appendix of this report.

This general procedure was used to analyze production Philip Morris (regular) cigarettes. The percentages of propylene glycol found were: 2.01, 2.06, 1.99, 2.04, 1.94, 1.69, 1.93, 1.77, 1.93. These values demonstrate a standard deviation of 0.1 percent in the cigarettes or a relative variation of 4%. The accuracy of the chromatographic analysis was established at $\pm 2.2\%$.

A study of the elution technique was made by collecting two milliliters of extract from each of five cigarettes; these extracts were analyzed and found to contain a total of 96 mg of propylene glycol. Two more milliliters were collected from each of these once extracted cigarettes, combined, and concentrated to one milliliter. This concentrate contained a total of 1.75 mg of propylene glycol. Another two milliliters from each cigarette contained a total of 0.14 mg.

These results indicated that 98.3% of the propylene glycol is extracted in the first two ml of methanol and 99.8% is extracted in four milliliters.

III. Conclusions

This method is rapid, accurate, and precise for the quantitative analysis of propylene glycol on cigarettes. However, the method cannot be extended to the analysis of glycerol without a temperature-programming unit.

IV. Recommendations

This method is recommended for the analysis of propylene glycol on cigarette filler but is not presently adaptable to the analysis of glycerol.

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V. Appendix

A. General Procedure

1. The cigarette is packed tightly into a tared glass tube (11 mm) that has been drawn to a small opening on one end. The weight of the cigarette is determined.
2. Methanol is added to the top of the tube and allowed to pass through the cigarette. Two milliliters of the extract are collected.
3. A ten microliter aliquot of the extract is applied to the top of a Ucon/Chromosorb column. The aliquot is added with a Hamilton micro-syringe.
4. The sample is chromatographed at 147°C with a carrier gas (Helium) flow of 86 ml/min.
5. The propylene glycol emerges after 2 minutes. The detector signal is attenuated at X10.
6. The area of the propylene glycol is measured using a planimeter and is compared to a standard curve of peak area versus concentration for propylene glycol chromatographed under the same conditions.

B. Column Preparation, Recorder, and Detector are identical to those referred to in the Completion Report for Project 21-0103 entitled "The Determination of Menthol and Triacetin in Cigarette Smoke."

C. Preparation of Standard Curve

1. A stock solution of propylene glycol in methanol is prepared and series dilutions of this stock solution are made to obtain solutions of 2 through 10 micrograms/microliter concentrations.
2. Ten microliter aliquots of these dilutions are applied to the column using a 50 microliter Hamilton syringe. The various sample sizes are thereby obtained.

The samples of extracts and stock solutions must be added in the same amount of solvent using the same syringe, or calibrated syringes.

The response of the detector changes with a change in solvent concentration.

Standard curves must be determined again when the cell filaments or filament current are changed.

D. Standard Curve for Propylene Glycol

The standard curve obtained by the method described above is included for illustration only.